



Primary Evaluator Samuel Ary, Chemist *Samuel Ary* Date: August 5, 2004  
Reregistration Branch II  
Health Effects Division (7509C)

Peer Reviewer Thurston Morton, Chemist *Thurston Morton* Date: August 9, 2004  
Reregistration Branch IV  
Health Effects Division (7509C)

Approved by Alan Nielsen, Branch Senior Scientist *Alan Nielsen* Date: August 10, 2004  
Reregistration Branch II  
Health Effects Division (7509C)

In the absence of signatures, this document is considered to be a draft with deliberative material for internal use only.

This data evaluation record (DER) was originally prepared under contract by Dynamac Corporation (20440 Century Boulevard, Suite 100; Germantown, MD 20874; submitted 07/21/2004). The DER has been reviewed by the Health Effects Division (HED) and revised to reflect current Office of Pesticide Program (OPP) policies.

#### **STUDY REPORT:**

45098405. Amoo, J.S. Magnitude of Residues of Thifensulfuron Methyl and Tribenuron Methyl in Cotton Following Application of Harmony Extra® Herbicide at Maximum Label Rates. Lab Project Number: AMR 4343-97. Unpublished study prepared by E.I. du Pont de Nemours and Company. 247 pages. March 21, 2000.

#### **EXECUTIVE SUMMARY:**

DuPont has included storage stability data in its submission of a cotton field study. Untreated samples of cotton seed and cotton gin trash (more appropriately referred to as cotton gin byproducts according to the U.S. EPA commodity definitions) were fortified with a mixture of thifensulfuron methyl and tribenuron methyl at 0.20 ppm each and stored frozen (ca. -20 °C) for up to approximately fourteen months. The results indicate that under these conditions, residues of thifensulfuron methyl were relatively stable in/on cotton seed and gin trash; refer to the other storage stability DER for the results concerning tribenuron methyl residues (S. Ary, D305958, MRID No.: 45098405, 8/10/04).

Samples of cotton seed and gin trash were analyzed for residues of thifensulfuron methyl using LC/MS (DuPont Method 1381). The validated limit of quantitation (LOQ) was 0.020 ppm, and the estimated limit of detection (LOD) was 0.006 ppm in/on cotton seed and gin trash. This method is adequate for data collection based on acceptable concurrent method recovery data.



## **STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:**

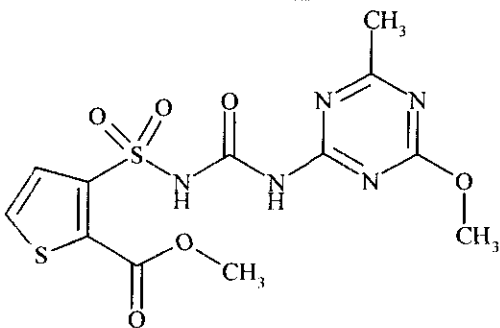
Under the conditions and parameters used in the study, the cotton storage stability data are classified as scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the forthcoming U.S. EPA "Summary of Residue Chemistry Data Evaluation Records for the Establishment of Tolerances for New Uses of Thifensulfuron Methyl on Canola, Flax, and Cotton" (DP Barcode: D301488).

## **COMPLIANCE:**

Signed and dated Good Laboratory Practice (GLP), Quality Assurance and Data Confidentiality statements were provided. No deviations from regulatory requirements were reported which would have an impact on the validity of the study.

### **A. BACKGROUND INFORMATION**

Thifensulfuron methyl is an herbicide registered for food/feed use on barley, oats, wheat, and soybeans. Thifensulfuron methyl is one of the active ingredients in DuPont Harmony® Extra (75% dry flowable [DF] formulation containing 50% thifensulfuron methyl and 25% tribenuron methyl) for postemergence use on barley, oats, and wheat, and preplant defoliant.

<b>Table A.1. Thifensulfuron Methyl Nomenclature.</b>	
Chemical structure	
Common name	Thifensulfuron methyl
Molecular formula	C <sub>12</sub> H <sub>13</sub> N <sub>5</sub> O <sub>6</sub> S <sub>2</sub>
Molecular weight	387.40 g/mol
IUPAC name	3-(4-methoxy-6-methyl-1,3,5-triazin-2-ylcarbamoylsulfamoyl)thiophene-2-carboxylic acid
CAS name	3-[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl]amino]sulfonyl]-2-thiophenecarboxylic acid
CAS registry number	79227-27-3
PC Code	128845
Current food/feed site registrations	Barley, oat, wheat, and soybean



**Table A.2. Physicochemical Properties of Thifensulfuron Methyl.**

Parameter	Value	Reference
Melting point/range	186 °C	S. Creeger, Memo, 11/1/84
pH	4.0 (slurry in water)	S. Creeger, Memo, 11/1/84
Density	1.49 g/mL	S. Creeger, Memo, 11/1/84
Water solubility	24 mg/L at 25 °C, pH 4.0 260 mg/L at 25 °C, pH 5.0 2400 mg/L at 25 °C, pH 6.0	S. Creeger, Memo, 11/1/84
Solvent solubility	11.9 mg/L at 25 °C in acetone 7.3 mg/L at 25 °C in acetonitrile 0.9 mg/L at 25 °C in ethanol 2.6 mg/L at 25 °C in ethyl acetate <0.1 mg/L at 25 °C in hexane 2.6 mg/L at 25 °C in methanol 27.5 mg/L at 25 °C in methylene chloride 0.2 mg/L at 25 °C in xylene	S. Creeger, Memo, 11/1/84
Vapor pressure	$2.7 \times 10^{-6}$ mm Hg at 25 °C	S. Creeger, Memo, 11/1/84
Dissociation constant	$pK_a = 4.0$	S. Creeger, Memo, 11/1/84
Octanol/water partition coefficient	$\text{Log}(K_{ow}) = 0.027$	S. Creeger, Memo, 11/1/84
UV/visible absorption spectrum	Not available	

## **B. EXPERIMENTAL DESIGN**

### **B.1. Sample Handling and Preparation**

Untreated samples of cotton seed and gin trash, obtained from a crop field trial study, were fortified with a mixture of thifensulfuron methyl and tribenuron methyl at 0.20 ppm each and stored frozen (ca. -20 °C). The fortification standard was prepared in methanol. Limited information concerning sample preparation and the storage vessels was included with the study; raw data were, however, provided. Fresh fortification samples were fortified at 0.20 ppm each analyte for each analysis set (time point); based on the analytical method description, these samples were fortified prior to extraction.

### **B.2. Analytical Methodology**

Samples of cotton seed and gin trash were analyzed by DuPont for residues of thifensulfuron methyl using LC/MS DuPont Method 1381, entitled "Analytical Enforcement Method for the Quantitation of Thifensulfuron Methyl and Tribenuron Methyl in Cotton Seed and Cotton Gin Trash by Liquid Chromatography/Electrospray Mass Spectrometry (LC/ESI-MS)". A description of the method was included in the submission; for a complete description of the method refer to the residue analytical method DER (S. Ary, D301488, MRID No.: 45098401, 8/10/04). This method is proposed for enforcement purposes in cotton.



## C. RESULTS AND DISCUSSION

Based on the concurrent method recovery data (see Table C.1), LC/MS Method 1381 is adequate for the determination of residues of thifensulfuron methyl in/on cotton seed and gin trash.

The results of the storage stability studies are presented in Table C.2. Residues of thifensulfuron methyl appear to be stable in/on cotton seed and cotton gin trash stored frozen for up to fourteen months. A graph of thifensulfuron methyl residue stability in these cotton matrices is presented in Figure C.1. Apparent residues of thifensulfuron methyl were below the method LOQ (less than 0.020 ppm) in/on one sample each of cotton seed and gin trash at each tested storage interval, except in/on one untreated cotton gin trash sample at day zero which bore residues of thifensulfuron methyl at 0.032 ppm. The registrant did not address the detectable residues in this cotton gin trash control sample.

Table C.1. Summary of Concurrent Recoveries of Thifensulfuron Methyl from Cotton Matrices.					
Matrix	Spike level (ppm)	Storage Interval (days)	Sample size (n)	Recoveries (%)	Mean
Cotton seed	0.20	0	2	105, 105	105
		18	2	95, 95	95
		35	2	90, 95	93
		88	2	100, 105	103
		423	2	95, 95	95
Cotton gin trash	0.20	0/1	2	70, 95	83
		16	2	80, 85	83
		35	2	85, 85	85
		87	2	90, 95	93
		422	2	80, 80	80



**Figure C.1. Graph of Thifensulfuron Methyl Stability in Cotton Matrices.**

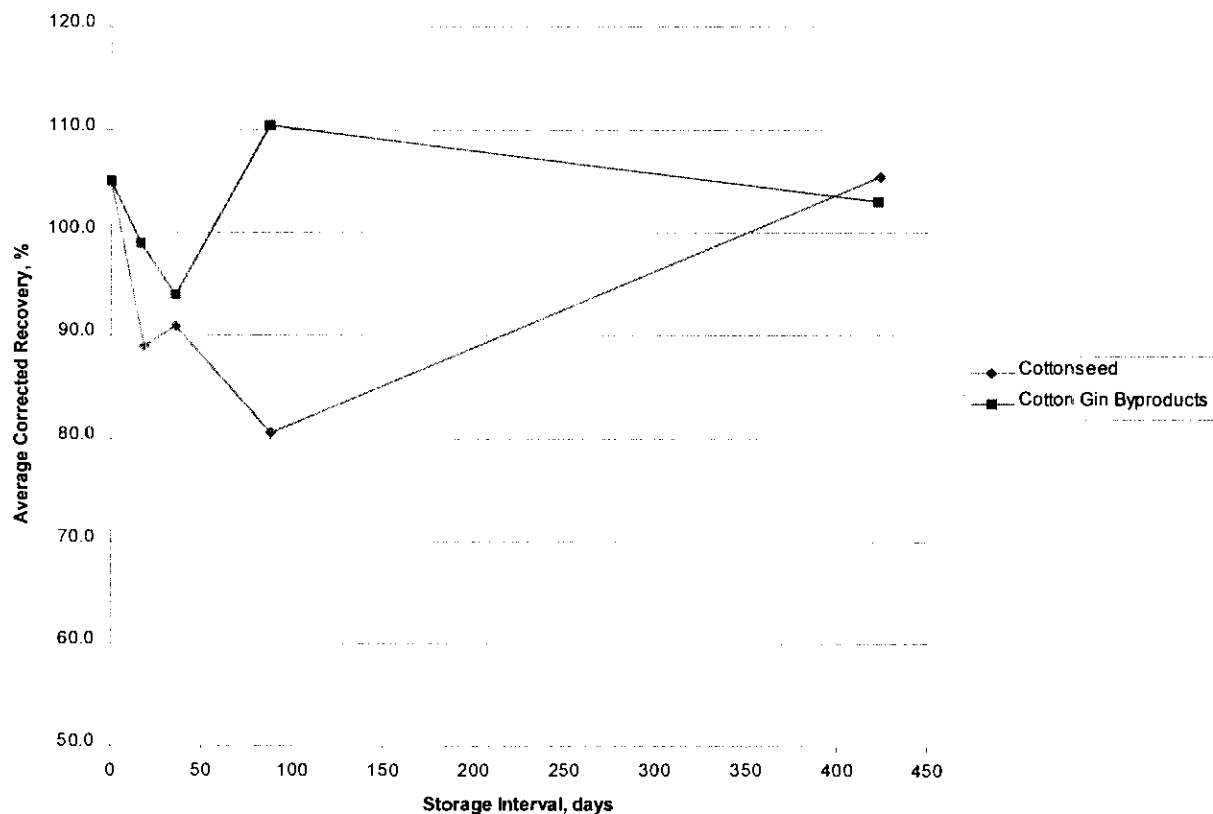


Table C.2. Stability of Thifensulfuron Methyl Residues in Cotton Seed and Cotton Gin Trash Following Storage at -20 °C.				
Commodity	Spike level (ppm)	Storage interval (days)	Recovered residues (ppm)	Corrected % recovery <sup>1</sup>
Cotton seed	0.20	0	0.21, 0.21	--
		18	0.17, 0.17	89, 89
		35	0.17, 0.17	91, 91
		88	0.16, 0.17	78, 83
		423	0.19, 0.21	100, 111
Cotton gin trash	0.20	0/1	0.17, 0.18	102, 108
		16	0.16, 0.17	96, 102
		35	0.16, 0.16	94, 94
		87	0.20, 0.21	108, 113
		422	0.16, 0.17	100, 106

1. Corrected for average concurrent method recoveries; see Table C.1.



#### **D. CONCLUSION**

The submitted storage stability results adequately demonstrate the stability of residues of thifensulfuron methyl in/on cotton seed and gin trash stored frozen for up to 423 and 422 days, respectively (ca. fourteen months). Acceptable methods were used for quantitation of residues in/on cotton seed and gin trash.

#### **E. REFERENCES**

Creeger, Samuel M. Thifensulfuron Methyl Memorandum. File Number: R032174. 49 pages. November 1, 1984.

Ary, Samuel. DER: Thifensulfuron Methyl Residue Analytical Method - Cotton. DP Barcode: D301488. MRID No.: 45098401. August 10, 2004.

Ary, Samuel. DER: Tribenuron Methyl Storage Stability - Cotton. DP Barcode: D305958. MRID No.: 45098405. August 10, 2004.

#### **F. DOCUMENT TRACKING**

RDI: S. Ary (8/5/04); S. Kinard (8/5/04); T. Morton (8/9/04); A. Nielsen (8/10/04)  
Petition Number: 0F6512  
DP Barcode: D301488  
PC Code: 128845



13544



R130182

**Chemical:** Thifensulfuron

**PC Code:**  
128845

**HED File Code:** 11000 Chemistry Reviews

**Memo Date:** 8/10/2004

**File ID:** DPD301488

**Accession #:** 000-00-0108

**HED Records Reference Center**  
8/23/2006

